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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(54) Title:</b> EPOXY-CONTAINING FOAMING COMPOSITIONS AND USE THEREOF  <b>(57) Abstract</b>  A foam composition and precursor thereto are disclosed. The foam precursor comprises: a) an epoxy, a modifying material, and a blowing agent, and: b) a carrier and an acid source.		

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TITLE

5 EPOXY-CONTAINING FOAMING COMPOSITIONS AND USE THEREOF

The subject matter herein claims benefit under 35 U.S.C. 111(a), 35 U.S.C. 119(e) and 35 U.S.C. 120 of U.S. Provisional Patent Application Serial  
10 No. 60/047,273, filed on May 21, 1997, entitled "A Room Temperature Foaming Composition"; and U.S. Provisional Patent Application Serial No. 60/079,205, filed on March 24, 1998, entitled "Novel Foaming Compositions and Methods For Making and Using the Compositions". The disclosure of the  
15 aforementioned Provisional Patent Applications is hereby incorporated by reference.

FIELD OF THE INVENTION

The instant invention relates to compositions, precursors thereof and  
20 methods of using the precursors to make foam and foam containing articles.

BACKGROUND OF THE INVENTION

Foams are employed in a wide range of commercial applications including automotive, construction, among other applications requiring thermal or sound insulation. In the automotive industry, foams are typically  
25 formed in situ, and can be used to fill cavities such as pillars and rocker panels, and to dampen sound transmission. In situ foam formation has typically been accomplished by using a polyurethane foam based on isocyanate chemistry. Certain polyurethane foam components and by-products thereof are believed to have an undesirable environmental impact. Consequently,  
30 there is a need in this art for a low-temperature foam which is cost-effective and substantially free of undesirable materials.

SUMMARY OF THE INVENTION

The instant invention solves problems associated with conventional  
35 foam formulations by providing foam compositions and precursors thereto which are not based upon polyurethane or isocyanate chemistry. The inventive compositions and precursors thereof reduce, if not eliminate, the presence of conventional undesirable compounds and by-products thereof while providing the benefits associated with conventional foams, e.g, sound dampening. The  
40 inventive compositions can be polyurethane and/or isocyanate free. By "free" it is meant that the inventive compositions before or after foaming contain less than about 10 wt.% polyurethane and/or isocyanurates, isocyanate, and in most

cases 0 wt.%. While the presence of such compounds does not adversely affect the reaction described below in greater detail, these materials can be obviated by employing the inventive formulations. The instant invention, therefore, provides a foam which can be used with or instead of urethane/isocyanate based foams and foaming systems.

One aspect of the invention relates to a method of contacting and in turn reacting the precursors of the composition at ambient conditions to produce a foam. This reaction produces a relatively large exotherm. The exotherm or heat released by the contact reaction was found to be sufficient to drive an endothermic blowing agent, thus creating a foam virtually instantaneously. Further, the exothermic reaction can cause a blowing agent entrapped within, for example, thermoplastic powders to expand thereby forming a foam.

Another aspect of the invention relates to a method of containing the foam during expansion by expanding the foam within a containment or control means. The control means confines the expanding foam and determines the direction of expansion. While any suitable control means can be employed, a polymeric bag or sack is desirable. The polymeric bag can be fabricated from a virtually unlimited array of materials and configured into any desirable shape.

The inventive foam can be employed in a wide array of end-uses. Examples of such uses include thermal insulation such as refrigeration, spray or rigid insulation for walls, marine foams, hot-water heaters, automotive sound dampeners, among others.

#### CROSS REFERENCE TO RELATED PATENT APPLICATIONS

The subject matter of the instant invention is related to Non-Provisional Patent Application Serial No. \_\_\_\_\_ (Attorney Docket No. OB001JP-1), filed on even date herewith in the name of Jeffrey Pachl et al., and entitled "Curable Sealant Composition". The disclosure of this Non-provisional patent application is hereby incorporated by reference.

#### BRIEF DESCRIPTION OF THE DRAWING

Figure 1 is a graphical representation of the foam reaction rate and temperature as a function of percent acid.

Figures 2A and 2B are a schematic drawings of assemblies that can be employed for dispensing the inventive foam within a defined cavity or area.

#### DETAILED DESCRIPTION

The inventive foam composition is obtained from foam precursors. The precursor comprises at least one epoxy, at least one carrier, e.g., a polyol, at least one hydrogen donor or an acid, e.g., phosphoric acid or a catalyst such as

a photoinitiator, optionally at least one thermoplastic, and at least one expansion or blowing agent, among other components. The foam precursor(s) can comprise a two component system (a so-called A and B side) that are contacted to produce a foam, or a single phase system that is activated in response to a source of energy, e.g., heat, UV light, among other energy sources. When a two component system is employed the epoxy and acid source are separated. An exothermic reaction between the epoxy and hydrogen donor or acid activates the expansion or blowing agent thereby producing a foam.

The epoxy comprises about 30 to about 50 wt% of the precursor. Examples of suitable epoxies comprise at least one member selected from the group consisting of bis-phenol a epoxy, epoxy modified elastomers, epoxy modified polybutene, epoxy modified polybutadiene, epoxy modified ethylene propyldiene rubber (EPDM), cycloaliphatic epoxy, mixtures thereof, among others. When a two component system is employed, the epoxy is located on the A-side, or otherwise prevented from reacting with the acid, e.g., the epoxy is separated from acid source.

The density, moisture and temperature resistance among other physical properties of the final foam product can be modified or tailored by adding a thermoplastic, thermoset, plastic or resinous material to the epoxy. While any suitable modifying material can be employed, examples of such materials comprise at least one member selected from the group consisting of ethylene vinyl acetate, polypropylene, polyethylene, rubber, phenoxy resin, phenolic, powdered wax, powdered epoxy, among others. About 1 to about 10 wt.% of modifying material can be added to the epoxy, e.g., about 2 wt.% of the precursor. The modifying material will normally comprise a powder having a particle size less than about 20 microns and a melting point from about 200 to about 270 C. The modifying material will become fluid and normally melt when exposed to the exothermic reaction temperature. When a two component precursor system is employed, these materials are normally combined with the epoxy or A side.

The carrier usually comprises about 20 to about 40 wt.% of the precursor, e.g., usually about 30 wt.%. The carrier serves to deliver an acid, expanding agent, catalyst, mixtures thereof, among others for contact with the epoxy. Examples of suitable carriers comprise at least one member selected from the group of polyester polyols, polyvinyl alcohol, water, among other carriers that are preferably miscible with the epoxy and mixtures thereof. When a two component precursor system is employed, these carrier materials are added to the acid side or "B" side component of the foam precursors. Depending upon the relative concentration of the components of the precursor, polyvinyl alcohol, hydroscopic polypropylene (as well as other suitable

materials) can be employed for absorbing steam or water generated by the exothermic reaction.

The hydrogen donor or acid usually comprises about 20 to about 30 wt.% of the precursor, e.g., about 5% to about 20% of precursor part B. Examples of suitable donors can comprise Lewis acids such as at least one member selected from the group consisting of sulfonic acids, phosphoric acid, citric acid, carboxylic acid, tannic and oxalic acids, mixtures thereof, among others. Particularly desirable results have been achieved by employing sulfonic and/or phosphoric acids. When a two component precursor system is employed, the acid corresponds to the B side of the foam precursors. If desired, the acid can comprises an acid functionally equivalent to the hydrogen donor released by a UV photoinitiator, e.g., replace a portion of the photoinitiator with its corresponding acid.

In one aspect of the invention, the hydrogen donor comprises a photoinitiator that becomes active when exposed to a source of energy. While any suitable hydrogen donor can be employed, an example of a suitable photoinitiator comprises a UV catalyst such as UVI 6974 (Union Carbide) that is described in greater detail in the aforementioned copending and commonly assigned U.S. Non-Provisional Patent Application (Attorney Docket No. OB001JP-1). When such a hydrogen donor is employed, the foam precursors can be utilized in a single phase system. For example, such a single phase system can be dispensed, exposed to a UV light source that causes the UV catalyst to generate an acid thereby permitting an epoxy based reaction to occur. The heat released by the exothermic epoxy reaction in turn activates an expansion or blowing agent, e.g., a hydrocarbon encapsulated within a thermoplastic, thereby producing a foam. While any suitable single or two phase system can be employed, normally a single phase system produces a foam that is thin relative to a two phase system.

The thermoplastic component of the foam precursor can comprise at least one member selected from the group consisting of acrylonitrile, polyethylene, phenolic, wax, EVA, polypropylene, EVA, GMA, acid modified polyethylene, polybutadiene, modified polyethylene blend (such as Bynel® supplied by DuPont Company), SIS blocked copolymers (such as Kraton® supplied by Shell Chemical), among other thermoplastic materials that can be dispersed in a foam precursor and have a melting point less than about the aforementioned exothermic reaction and mixtures thereof. Normally, the thermoplastic component of the precursor will comprise about 10% to about 15 wt.% of the precursor. The thermoplastic component can possess any desirable configuration or particle size. In some cases, the thermoplastic component can form a film or skin upon an exterior surface of the foam thereby improving the resistance of the foam to fluids, e.g., water, gasoline, among other fluids.



In one particularly useful aspect of the invention, a blowing agent is combined with or encapsulated within a thermoplastic particle or powder, e.g., a hydrocarbon encapsulated within an acrylnitrile shell known as Expancel® that is supplied by Nobel Industries. When a two component precursor system is employed, the shells are normally combined on the B side along with the carrier. These shells can, however, be combined with the A side or in a single phase system so long as the composition of the shells is not substantially affected by the epoxy, e.g, the acrylnitrile shells may be soluble within the epoxy. For example, the shells can be fabricated from polyethylene, nylon, EVA, polypropylene, among other materials not soluble in the epoxy component, and mixtures thereof could be present in the epoxy component of a two phase precursor system. When the A and B sides are contacted (or in the case of a single phase system exposed to an energy source), the epoxy reacts with the hydrogen or acid thereby releasing heat and causing the expansion agent within the shells to foam. The foam can be characterized by a composite wherein the epoxy reaction product (including of the aforementioned modifying materials) forms a matrix that embeds the expanded shells. Depending upon the physical characteristics desired in the foam, the shells can be open or closed cells.

The aforementioned blowing agent can comprise at least one member selected from the group consisting of water, hydrazide, diphenyloxide-4,4-disulphohydrazide, carbonamide, azocarbonamide, hexamethylene diamine carbamate, sodium bicarbonate, carbon dioxide, hydrofluorocarbons such as difluoroethane, tetrafluoroethane, among others; hydrocarbons such as butane, propane; mixtures thereof, among others. Normally, the expansion or blowing agent comprises about 5 to about 40 wt.% of the foam precursors.

The foam characteristic can also be modified by adding one or more filler materials. Conventionally used filler materials comprise at least one of talc, mica, magnesium silicate, mixtures thereof, among others. If desired calcium carbonate can be added to the foam precursor for increasing the hardness and density of the resultant foam. When included the filler comprises about 5 to about 35 wt.% of the foam precursors.

An example of a combined A and B side precursor composition is set forth in the following Table.

35

TABLE

Chemical Name	Trade Name	Supplier	Wt. %	Equivalent
Cycloaliphatic Epoxy	Uvacure 1500	UCB Radcure	30 - 50	Sartomer SARCAT K126
Polyester Polyol	Tone 0301	Union Carbide	20 - 30	-----
Phosphoric Acid	Phos. Acid	J.T. Baker	5 - 15	commodity

40

Thermoplastic  
Microspheres

Expancel Nobel Industries 5 - 15

H. M. Royal

5

The precursors can be combined by using any suitable apparatus that imparts an amount of shear sufficient to obtain a substantially homogenous precursor. Examples of suitable apparatus comprise hand mixing, static tube mixtures, the structures described illustrated by Figures 2A and 2B (described below in greater detail), among other conventional apparatus. Normally, the samples are mixed for about 2 to about 10 seconds depending upon the composition and mixing environment, e.g., a 1:1 A:B composition can be mixed for about 5 seconds in a static tube mixer.

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The inventive foam can be formed by contacting the precursors under conditions sufficient to cause the aforementioned exothermic reaction which causes the blowing agent to expand and in turn form a foam. Prior to being contacted, the precursors can be preblended and stored in separate containers. That is, a Part A or first precursor mixture is typically obtained by combining the epoxy and modifying material, e.g. polyvinyl alcohol and polypropylene, and a Part B or second precursor mixture can be obtained by combining the carrier, e.g., a polyol, hydrogen donor/acid and thermoplastic, e.g., encapsulated blowing agent. Part A and Part B can be combined to obtain a foam by using conventional foam manufacturing equipment. For example, Part A and Part B can be contacted as two high pressure streams within a mixing chamber of an external mix-head. While heat can be added to the precursors, the reaction between "A" and "B" can occur under ambient conditions. The ratio of "A" to "B" normally ranges from about 1:1 to about 5:1.

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The pH of the A side (or epoxy) is normally about 6 to at least about 8. The pH of the B side of foam precursor comprising an acid and a carrier is normally about 0.5 to about 4, e.g., the pH of phosphoric acid when mixed with polyol. Normally, the pH prior to reaction with part A is about 1.6 The composition and concentration of the foam precursors can be modified to achieve a predetermined reaction rate e.g., by tailoring the concentration of the acid. The affects of the pH or acid concentration of the B side are better understood by reference to Figure 1 which illustrates the affects upon the composition demonstrated in Example 9. Referring now to Figure 1, Figure 1 is a graphical representation of % acid in the precursor versus foam reaction time and temperature. Figure 1 illustrates that as the acid concentration increases the reaction temperature increases and the reaction time decreases. Figure 1 also illustrates that the precursor can be selected to a predetermined reaction time/temperature. For example, by selecting a higher reaction temperature (higher acid concentration) a wider range of modifying materials

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can be employed whereas by selecting a slower reaction time (lower acid concentration) the foam has easier handling characteristics.

The viscosity of a foam precursor can be tailored to enhance the resultant foam characteristics. The viscosity of the "A" side or epoxy component of the foam precursor is normally controlled, for example, so that a modifying material, e.g., a plastic powder, becomes or remains dispersed within the "A" side precursor. While any suitable viscosity control agent can be employed desirable results can be achieved by using a solid polymer (in particulate form) to produce a foam precursor gel. Examples of suitable solid polymers comprise at least one member selected from the group consisting of waxes, polyethylene, EVOH, PVOH, fluoropolymers and dispersions thereof such as polytetrafluoroethylene (supplied as Teflon® by the DuPont Company), among others. The viscosity control agent can range in particle size of about 20 to 50 microns, e.g., less than 325 mesh. An example of a controlled viscosity composition comprises about 5 to about 10 wt.% solid epoxy, about 5 to about 15 wt.%, powdered polyethylene and about 25 to about 30 wt.% blowing agent. In addition to viscosity, the characteristics of the foam can be tailored by varying the temperature, pressure, foam pH, foam density, among other parameters known to those skilled in this art. Also, the 'A' side of the system can be thickened into a gel by the addition of a surfactant such as any commercially available liquid detergent or titanate such as Kenrich KRTTS, e.g., about 0.5 to about 3 wt.% surfactant. This enables a more complete rheological control, included insuring the homogeneity of the system.

In one aspect of the invention, the foam comprises a single phase system, e.g., only an "A" side mixture. An example of such a system comprises an epoxy, a polyol, thermoplastic spheres, modifying materials, phenoxy, polypropylene, mixtures thereof, among other components. This system can be heat activated. In other words the system expands by being exposed to elevated temperature, e.g., about 125C. If desired the single phase foam system can be initiated by employing a photo-initiator instead of or in conjunction with an elevated temperature. Examples of such initiators comprise at least one member selected from the group consisting of Union Carbide UV1 6974 among others. Normally, the amount of such an initiator corresponds from 5 to about 15 wt% of the foam precursor. More details regarding photoinitiators can be found in "Photopolymerization Behavior of Several Cationic Photoinitiators in Catatonically Cured Resin Systems" by Edward Jurczak; that is hereby incorporated by reference. This single phase system is especially useful when applied upon a substrate by being sprayed. For example, the single phase system can be sprayed upon an automotive subassembly for reducing the amount of sound transmission to the interior of the car. In a further example, the single phase system can be sprayed upon a

first component, e.g., a plastic facia, exposed to UV to cause foaming and affixed upon a second component, e.g, metal support member, wherein the foam functions to reduce vibrations between the components.

5 A composite foam structure can be obtained in accordance with the instant invention. A structural modifier such as fibers, particles, rods, tubes, powders, mixtures thereof, among others, can be incorporated as a component of the foam precursor. The structural modifier can be employed for tailoring the chemical and/or physical properties of the resultant foam. Examples of  
10 suitable structural modifiers, normally as chopped fibers or powders, can comprise at least one of nylon, carbon, carbonates, polymers such as polyethylene and polypropylene, graphite, Kevlar®, Dyneon, ceramic, fiberglass, mineral fillers, e.g., mica, metals, among other materials. The amount of such structural modifiers normally comprises about 1 to about 30 wt.% of the uncured foam precursor.

15 Any suitable commercially available foam production equipment can be employed for mixing and dispensing the inventive foam precursors to obtain the inventive foam. An example of such equipment comprises ECONO-MIX supplied by Kirkco Corporation, Monroe, N.C. Another example comprises using a DO-PAG pump in combination with a Albeon static mix head. If  
20 desired, the inventive foam can be expanded within a cavity, e.g., an automotive A pillar, by employing a dispensing apparatus having a replaceable/disposable static mix head. That is, the static mix head can comprise a replaceable plastic tubing having a center piece with a helix or vortex configuration, that is connected to a pump discharge flange and inserted  
25 into the cavity for foaming the precursors. Another static mix head design has a valve type of arrangement that is illustrated in Figures 2A and 2B. Referring now to Figure 2A, Figure 2A illustrates a one-way valve type of arrangement wherein the foam or precursors thereof are introduced or injected via a one-way valve 1 (commonly known as a zerk) that is positioned within a cap 2.  
30 Valve 1 can also include a flap or secondary valve 1A that prevents foam from escaping by reverse flow through valve 1. The cap 2 seals or defines one end of a cavity being filled with foam. The cap 2 can include hooks or locking tabs 3 for securing the position of cap 2, e.g, within the so-called A pillar of an automobile thereby permitting foam to be dispensed within the automotive  
35 cavity in a controlled manner. Normally, one end of the valve 1 is connected to a mixing zone 4 such as the aforementioned static mixers having helical vanes 5. After traveling through the valve 1 and static mixer 4, the foam is released into the cavity to be filled with foam. The area and direction into which the foam expands can be control and/or defined by using a containment  
40 means such as a polymeric bag 6 (the containment means is described below in greater detail).

Referring now to Figure 2B, this type of arrangement provides a longer mixing time for the foam precursors before the foam is released into the cavity to be filled. The arrangement illustrated in Figure 2B can also be employed as a cap 10 to seal or define one end of the cavity to be sealed. After delivering the foam precursors, a mix head 11 or previously described valves (4 and 5 of Figure 2A). The foam precursors travel through mix head 11 and are released at the opening defined at 12 as a foam. The opening 12 can also be within the aforementioned containment means. The caps 1 and 10 can remain associated with the foam product within the cavity. By using such a replaceable mix head, any problems associated with clogged mix heads are avoided. Two pressure streams can also be employed, to converge in a mix chamber or cavity to be foamed and mix action occurs without use of additional mixing apparatus. In addition to the foregoing, the inventive foam composition can be injected, extruded, shaped, sprayed, molded, among other conventional processes in order to obtain a desirable foam article.

While the above description emphasizes particular foam compositions, the inventive compositions (and precursors thereof) can include additives such as dyes, fillers, surfactants, pigments, nucleating agents, among other conventional employed foam additives. If desired a pH indicator can be added to the precursor in order to provide a visual detection means for a reaction product. An example of a suitable pH indicator comprises at least one member selected from the group consisting of methyl red, methyl blue, chlorophenol red, bromothymol blue. That is, as the foam precursor react, e.g., acid-epoxy, the acid is consumed thereby changing the pH and causing the pH indicator to change color.

If desired, the inventive composition can be laminated or joined with other articles, e.g., laminated onto metal foil, mylar, fiberboard etc.. The inventive composition can also be expanded within a control or containment means or bag having a predetermined shape thereby forming a foamed article that replicates the bag, e.g., refer to U.S. Patent Nos. 4,269,890 (Breitling), 4,232,788 (Roth), 4,390,333 (Dubois); the disclosure of each of which is hereby incorporated by reference. When expanding the foam into a bag, the previously described valves illustrated in Figures 2A and B, those supplied commercially by Inflatable Packaging as part no. IP04, or any other suitable delivery means can be employed at the opening in the bag in order to control introduction of the foam into the bag. For example, a bag replicating a cavity such as an automotive cavity or any other desirable configuration unrolls or expands into the cavity as foam is introduced into the bag via the valve. If desired, the bag may comprise or be coated with a heat sensitive adhesive wherein the heat generated by the exothermic foam reaction activates the adhesive. The adhesive can permanently affix the foam containing bag at any desirable location. The bag can also include predetermined areas having

weakened seams or perforations that are designed to rupture as the foam expands thereby directing the expanding foam. Similarly, the bag composition can be selected such that the bag melts when exposed to the foam. The melting bag can direct the expanding foam, form a coating upon the foam, function as an adhesive, among other utilities. Further, a plurality of bags can be employed wherein one bag is surrounded by another bag. The inner and/or outer bag can possess the aforementioned predetermined properties. Furthermore, the bag can comprise areas having distinct chemical and/or physical properties, e.g., a bag comprising one sheet of polyethylene heat sealed around its periphery to a sheet comprising polybutadiene. At least a portion of the bag can be fabricated from one or members selected from the group consisting of polyethylene, polyester, vinyl, nylon, Surlyn®, ethylene vinyl acetate, styrene-isoprene-styrene, styrene-butadiene-styrene or other blocked copolymers, polybutadiene, among other plastic materials with melt points corresponding to temperature range of reaction, polyamide, modified EVA's, modified polyethylene, modified polybutadiene, GMA, SBR, among other plastic materials suitable for bag or bladder construction and seaming capability. The bag or containment means can be utilized with a wide range of foam compositions in addition to the previously described epoxy containing foams. Examples of foams that can be expanded into the previously described containment bags or means comprise at least one of epoxy amine, acrylic, phenolic among others.

The following Examples are provided to illustrate not limit the scope of the invention as defined in the appended claims. Unless indicated otherwise, commercially available apparatus and materials were employed in these Examples.

#### EXAMPLE 1

A foam product was produced by mixing a 2-part system (Part A and Part B) through a conventional foam production apparatus comprising a static mixer that was manufactured by Albion (Model No. 535-1 or equivalent). The constituents of the foam were maintained in two separate supplies of materials, Part A and Part B.

Part A comprised a blend of the epoxy and the thermoplastic microspheres (a blowing agent) in ratio of 30 parts to 15 (100 parts total). Part B comprised a blend of the phosphoric acid and the polyol in a 30 part to 50 part ratio (also 100 parts). The feed ratio of Part A to Part B to the mixer head was 1:1. The pH of the Part B was about 1.6 prior to reaction with Part A.

A pressurized flow through the mixing chamber produced a polymer which rapidly expands and released an amount of exothermic heat sufficient to produce a foam.

## EXAMPLE 2

The process of Example 1 was repeated with the exception that the ratio of epoxy to thermoplastic microspheres in Part A was 2:1, and the ratio of phosphoric acid to polyol in Part B was 3:5. The feed ratio of Part A to Part B to the mixer head was 3:1.

## EXAMPLE 3

The process of Example 1 was repeated with the exception that the Part A and Part B components were mixed together by hand (instead of using the static mixer).

## EXAMPLE 4

This example demonstrates the formation of a composite foam. The process of Example 1 was repeated with the exception that about 5 wt.% polytetrafluoroethylene powder (TEFLON® supplied by the DuPont Company) was added to the Part A composition. Part A and Part B were contacted in the manner described in Example 1. A composite foam was recovered wherein the composite foam had greater flexibility or pliability in comparison the foam obtained by the process of Example 1.

## EXAMPLE 5

A two phase system was used to produce a foam. Part A was composed of epoxy and microspheres in a 2:1 ratio (67 % epoxy, 33.3% microspheres) by weight.(for best results, the mix should be used within in 4-8 hours of mixing since epoxy can dissolve certain spheres ). Part A was hand stirred to a smooth consistency.

Part B was composed of Polyol (Tone 301) and Phosphoric acid (10%) by weight). The acid was blended into the polyol. Part A to Part B ratio of 1:1 was contacted in a static tube mixer (a commercially available tube having internal vanes that mix any material passing through the tube) and produced a foam. The ratio of A to B can be from 1:1 to 4:1 depending on acid concentration.

## EXAMPLE 6

A two phase system was used to produce a foam. Part A comprised an epoxy (Radcure 1500). Part B was comprised of a polyol (50 wt.% - tone 301), phosphoric acid diluted with water (approximately 50% acid in a commercially available solution) at 20%, and 30% microspheres. The spheres were hand stirred into the polyol to a smooth consistency. The acid mixture was blended by hand stirred into the sphere- polyol mix. Part A to Part B ratio

of 1:1 was contacted in a static tube mixer and produced a foam. The ratio of A to B can be from 1:1 to 4:1 depending on acid concentration.

#### EXAMPLE 7

5 A two phase system was used to produce a foam. Part A comprised an epoxy (Radcure 1500) Part B comprised polyol (tone 301), polyvinyl alcohol and water blend (PVOH : H<sub>2</sub>O 3:1 blend that corresponded to 20% of the polyol) and microspheres 30% by weight of polyol and acid can be 10% of total 'B' mixture. The spheres were hand stirred into the polyol to a smooth  
10 consistency. The PVOH and water are hand stirred. The PVOH/water solution temperature was 140 °F. The PVOH blend was added to the polyol by hand stirring. The acid was hand stirred into the sphere-PVOH- polyol mix. Part A to Part B ratio of 1:1 was contacted in a static tube mixer and produced a foam. The ratio of A to B can be from 1:1 to 4:1 depending upon acid  
15 concentration.

#### EXAMPLE 8

20 A two phase system was used to produce a foam. Part A comprised an epoxy (Radcure 1500) and a phenoxy resin (Paphen 200 that corresponded to 25% of part A, epoxy is 75% of part A). Part B comprised 45 % polyol (tone 301), 23.5% polyvinyl alcohol (Air Products 203S) and 23.5% microspheres. Phosphoric acid was 10% of Part B. Spheres are hand stirred into the polyol  
25 to a smooth consistency. The PVOH, microspheres, and polyol are blended by hand stirring. The phosphoric acid was hand stirred into the sphere-PVOH- polyol mix. Part A to Part B ratio of 1:1 was used contacted in a static tube mixer to produce a foam. The A to B ratio can range from 1:1 to 4:1 depending on acid concentration.

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#### EXAMPLE 9A

A two phase system, namely a Part A and Part B, was used to produce a foam. Part A comprised an epoxy (Radcure 1500) 60 wt%, polypropylene powder (Equistar FP 800-00) 20 wt%, polyvinyl alcohol (Aerosil 203S) 20  
35 wt%. Part B comprised polyol (Tone 301) 60 wt% and microspheres 30%. Phosphoric acid was 10%. Spheres are hand stirred into the polyol until a smooth consistency was obtained. The microspheres and polyol are blended by hand stirring. The phosphoric acid was hand stirred into the microspheres and polyol mix. A Part A to Part B ratio of 1:1 was used and contacted in a static  
40 tube mixer to produce a foam. The A to B ratio can, however, range from 1:1 to 4:1 depending on acid concentration.



## EXAMPLE 9B

This Example employed a two phase system wherein the Part A comprised a gell. A two phase system, namely a Part A and Part B, was used to produce a foam. Part A comprised an epoxy (Radcure 1500) 59 wt%, polypropylene powder (Equistar FP 800-00) 20 wt%, polyvinyl alcohol (Aerosil 203S) 20 wt% and surfacant (gelling agent) at 1 wt%. Part B comprised polyol (Tone 301) 60 wt% and microspheres 30%. Phosphoric acid was 10%. Spheres are hand stirred into the polyol until a smooth consistency was obtained. The microspheres and polyol are blended by hand stirring. The phosphoric acid was hand stirred into the microspheres and polyol mix. A Part A to Part B ratio of 1:1 was used and contacted in a static tube mixer to produce a foam. The A to B ratio can, however, range from 1:1 to 4:1 depending on acid concentration.

## EXAMPLE 10

A bag or containment means approximately 8x8 inches having a one-way valve located on one end of the bag was constructed from two sheets of high density polyethylene film. The seams of the bag were designed to rupture at specific locations, which directs foam expansion into cavity area adjacent to weak seams. The sheets were joined by heating on a TEW Electric Heating Company Ltd sealing apparatus. The seams were selectively reinforced by double sealing or weakend to provide multiple points for foam direction from the same bag. The foam composition demonstrated by Example 9 was introduced into this bag. As the foam expanded, the foam escaped from the bag through the relatively weak seams.

## EXAMPLE 11

A Bag or bladder composed of each of polyethylene, ethylene vinyl acetate, polybutadiene were fabricated by using the apparatus described in Example 10. The foam of Example 9 was introduced into these bags. The bags, having a melting point less than the exothermic reaction temperature of the foam, failed and released the foam.

## EXAMPLE 12

A bag or bladder composed of each of modified EVA (Bynel®), modified polyethylene (Primcor® supplied by Dow Chemical Company), modified butadiene, glycidal methacrylate (GMA) were fabricated by using the apparatus of Example 10. The foam of Example 9 was introduced into these bags. The heat released from the exothermic reaction of the foam caused the bags to melt. The melting bag material adhered to the foam thereby modifying the surface of the foam. The melting bag also adhered the foam to any surrounding surfaces or articles.

## EXAMPLE 13

A bag or bladder composed of each of polypropylene, polyethylene, woven nylon mesh, aluminized fiberglass mesh was fabricated by using the apparatus of Example 10. Each of the bags was further processed to possess multiple perforations (25- 100 holes/in.). The foam of Example 9 was introduced into each of these bags. The perforations allowed the foam to escaped in controlled quantities while also generally retaining the shape of the bag.

## EXAMPLE 14

Two bags or bladders, namely an inner and outer bag were fabricated by using the apparatus of Example 10. The inner bag comprised modified butadiene and the outer bag comprised high density polyethylene. The inner bag was placed within the outer bag and the outer bag was sealed. The foam of Example 9 was introduced into the inner bag. Inner bag or bladder melted during the foam reaction. The inner bag was of sufficient size to contain the required amount of mixed foam precursors to fill the out bag. Outer bag construction was of material and size to contain reaction within the cavity.

## EXAMPLE 15

The insertion loss or sound dampening characteristics of the foam produced in accordance with Example 9A was tested in accordance with Society of Automotive Engineers (SAE) J 1400. The sample size was 3x3x10 inches and placed within an E-coated metal channel. An increase in insertion loss corresponds to an increase in sound dampening properties that in turn corresponds to less noise within the passenger compartment of an automobile.

	FREQ. (Hz)	INSERTION LOSS (dB)
30	125	12.5
	160	10.6
	200	11.4
	250	12.0
	315	24.5
35	400	35.4
	500	46.8
	630	38.4
	800	40.1
	1000	45.7
40	1250	45.1
	1600	49.6
	2000	49.2

	2500	50.1
	3150	50.9
	4000	55.5
	5000	58.7
5	6300	59.2
	8000	64.2

These data illustrate the desirable sound absorbing characteristics of the inventive foam compositions.

10 A skilled person in this art would understand that these Exemplary processes can be modified by manipulating process variables such as time and temperature of each aforementioned mixing step, mixing rate (RPM), time under vacuum, UV light exposure, and level of vacuum (mm Hg) as well as operating a continuous process. While the above Examples illustrate a batch process a skilled person in this art after having reviewed and understood the  
15 instant disclosure, would be capable of manipulating the aforementioned process variables to tailor the instant composition for a virtually unlimited array of product applications.

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THE FOLLOWING IS CLAIMED:

- 5           1. A foam precursor comprising:  
            (a) an epoxy, a modifying material, and a blowing agent, and;  
            (b) a carrier and an acid source.
- 10           2. The foam precursor of Claim 1 wherein said (a) comprises a  
            cycloaliphatic epoxy; at least one member selected from the group  
            consisting of polypropylene, polyethylene and polyvinyl alcohol; and a  
            butane blowing agent.
- 15           3. The foam precursor of Claim 1 wherein said (b) comprises a  
            polyester polyol and phosphoric acid or a photo-initiator.
4. The foam precursor of Claim 1 wherein further comprising a  
            blowing agent encapsulated within a thermoplastic shell.
- 20           5. A foam obtained by contacting (a) and (b) of Claim 1.
6. A method for making a foam comprising:  
                preparing a first mixture comprising an epoxy, a thermoplastic  
                and a blowing agent,  
25              preparing a second mixture comprising a carrier and an acid  
                source,  
                contacting said first and second mixtures under conditions  
                sufficient to cause an exothermic reaction which is sufficient to  
                expand said contacted mixtures to form a foam, and;  
30              recovering the foam.
7. The foam precursor of Claim 2 or 3 wherein the thermoplastic  
            comprises spheres containing a butane blowing agent.
- 35           8. An "A" side foam precursor consisting essentially of an epoxy,  
            polyvinyl alcohol and a plastic powder.
9. A "B" side foam precursor consisting essentially of a polyol and  
            an acid.
- 40

10. The foam precursor of Claim 1 further comprising at least one member selected from the group consisting of nylon, carbon, polyethylene, polypropylene, polytetrafluoroethylene and graphite.

5 11. The foam of Claim 4 wherein said foam comprises a composite containing at least one of fibers, particles, rods, tubes and powders.

12. The foam precursor of Claim 1 further comprising an effective amount of at least one photoinitiator.

10 13. A foam composite comprising the foam of Claim 1 or 2 wherein said foam is at least partially in contact with at least one member selected from the group consisting of polyethylene, polyester, vinyl, ethylene vinyl acetate, nylon, ethylene vinyl acetate, styrene -isoprene-  
15 styrene, styrene- butadiene -styrene or other blocked copolymers, polybutadiene, polyamide, modified EVA's, modified polyethylene, modified polybutadiene, GMA, SBR and mixtures thereof.

14. The method of Claim 12 wherein at least a part of said  
20 contacting occurs with in a containment means.

15. The method of Claim 13 wherein the containment means comprises a cavity defined within at least one material selected from the group consisting of polyethylene, polyester, vinyl, ethylene vinyl  
25 acetate, nylon, ethylene vinyl acetate, styrene -isoprene- styrene, styrene- butadiene -styrene or other blocked copolymers, polybutadiene, polyamide, modified EVA's, modified polyethylene, modified polybutadiene, GMA, SBR and mixtures thereof.

30 16. The foam or foam precursor of Claim 1, 8 or 9 further comprising a surfactant.

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FOAM REACTION TEMPERATURE / TIME

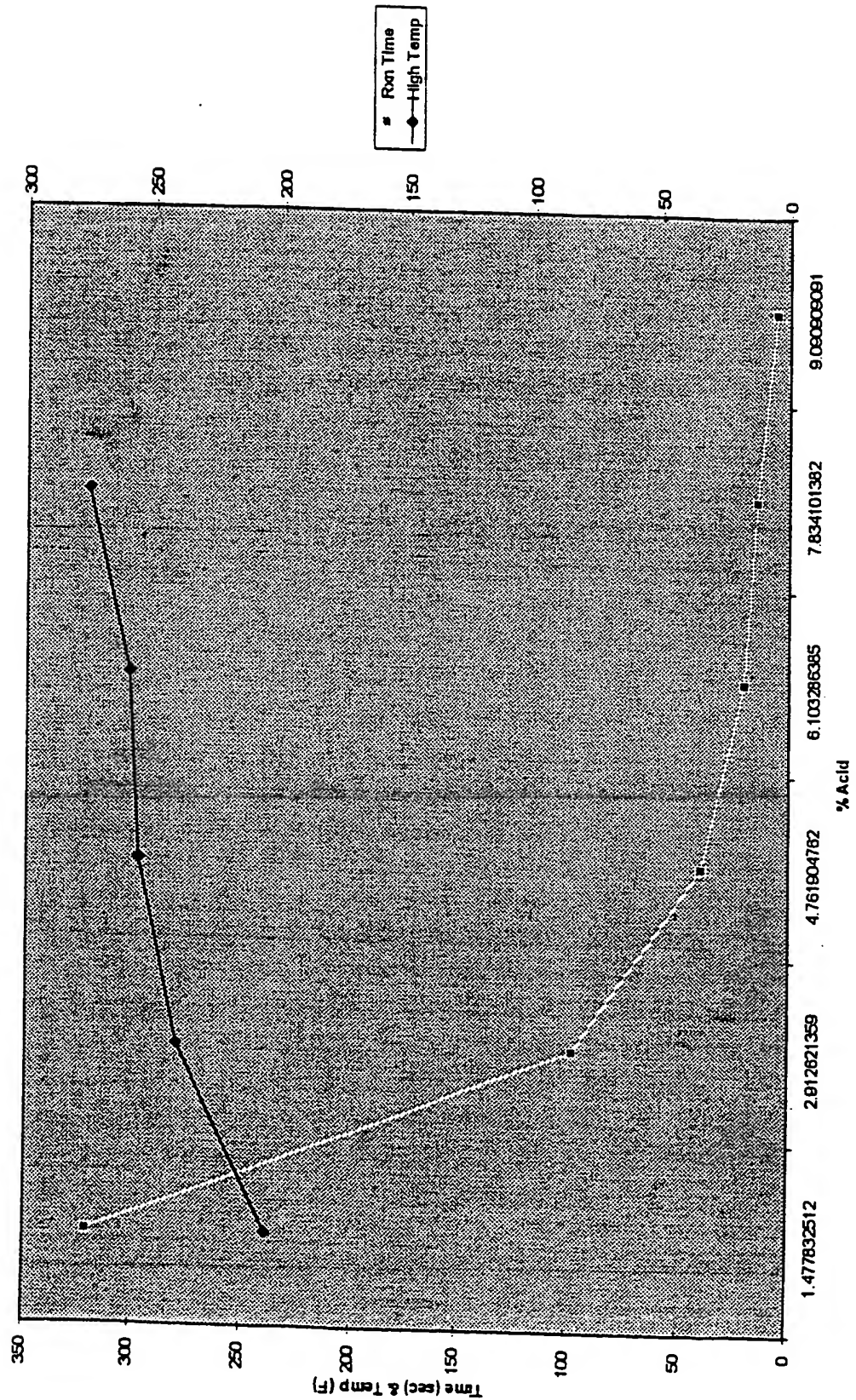
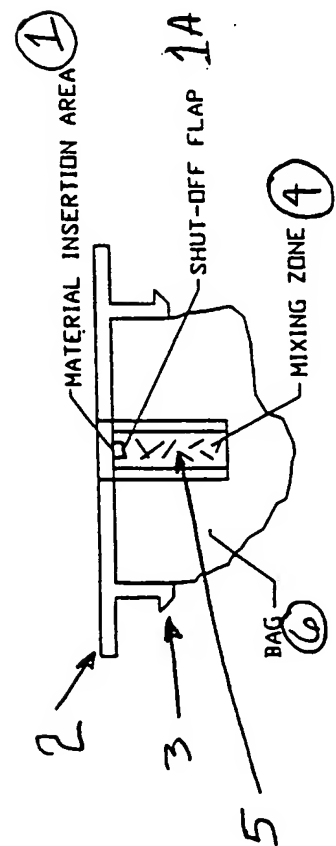


Fig 1

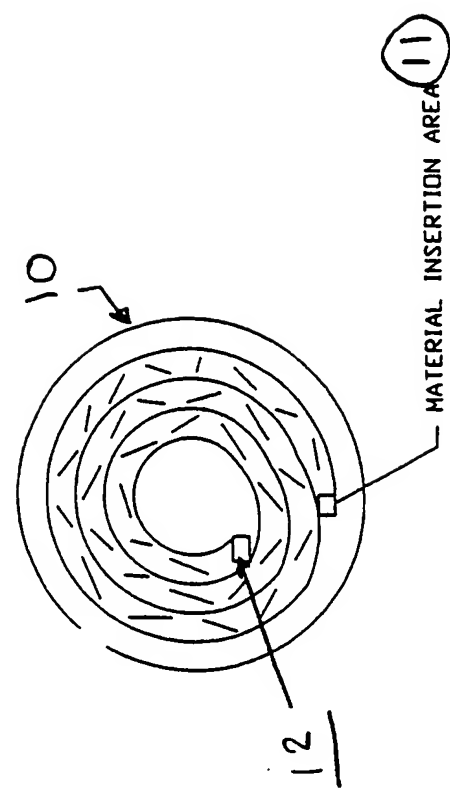
Fig 2

2A

2B



CENTER MIXING ZONE



CIRCULAR MIXING ZONE

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 98/10331

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 6 C08J9/00 C08J9/32

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 012 593 A (DESAI POLYMER DEV) 25 June 1980 see page 3, line 26 - page 4, line 9 see claims	1, 10, 11, 16
X	US 5 183 583 A (WISHNESKI TODD W ET AL) 2 February 1993 see claim 1	9
A	GB 1 485 995 A (BOLIDT LICENSE NV) 14 September 1977 see page 2, column 57-60 see page 3, line 106-123 see page 6; example 6 see claims	1
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

\* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

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Form PCT/ISA/210 (second sheet) (July 1992)



## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 98/10331

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Section Ch, Week 7621 Derwent Publications Ltd., London, GB; Class A14, AN 76-38795X XP002074707 & JP 51 041 028 A (FUJIKURA KASEI KK) see abstract ----	1
A	DATABASE WPI Section Ch, Week 9137 Derwent Publications Ltd., London, GB; Class A21, AN 91-270311 XP002074708 & JP 03 177 439 A (MITSUI TOATSU CHEM INC) see abstract -----	1

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Information on patent family members

International Application No

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